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Statistical Considerations for Adiabatic Compression Testing

Citation

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ABSTRACT

ASTM G74 has been used for many years to evaluate nonmetallic materials and components for oxygen service. When originally published in 1982, this standard considered a "passing" result to be zero ignitions of a material out of 20 samples tested. However, researchers have recognized that the originally prescribed methodology results in a cumulative binomial confidence of about 36 % for a passing result. As a result, the low confidence for a passing result could be potentially misleading when results are used to qualify materials or components for oxygen service, unless the data is analyzed through available statistical approaches. This paper summarizes research performed to evaluate the statistical aspects of gaseous fluid impact testing so that ignition probabilities can be considered in the test methodology. Data derived by the test method are evaluated by a logistic regression approach in order to describe the behavior of the materials being tested and to compare different materials or test conditions. Therefore, the statistical aspects of the test are shown to be crucial to understanding and applying the data obtained. This paper demonstrates that the ASTM G74 test and all international tests of a similar nature because all use the same test embodiment and are inherently probabilistic and subject to variability that seems random without application of appropriate statistical analysis. However, meaningful results can be developed when the appropriate statistical tools are utilized. Logistic regression analysis is only one available method to

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analyze binomial data (ignition/no-ignition); but it is a powerful tool that can help to bring clarity to the trends in data that are obscured by sometimes seemingly random behavior.

Keywords

gaseous fluid impact, adiabatic compression, oxygen, oxygen compatibility, oxygen qualification, test severity, logistic regression, material or component qualification, material or component ignition, ASTM G74

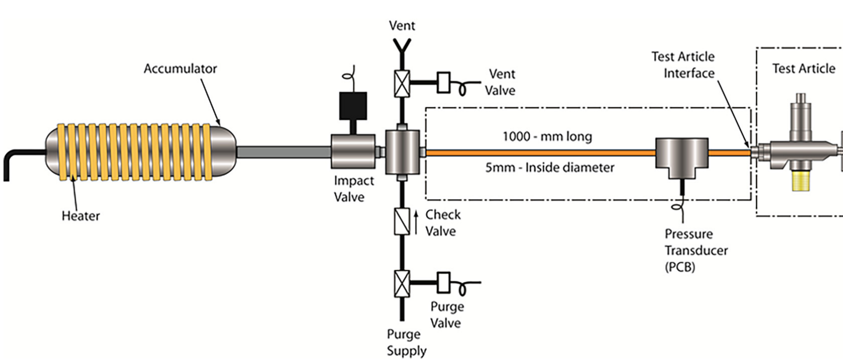
Introduction

Gaseous fluid impact testing, commonly referred to as adiabatic compression testing, is widely used for ranking a non-metallic material for its sensitivity to ignition in gaseous oxygen according to ASTM G74 [1]. This testing is also used for qualifying flow control equipment (valves, regulators, flexible hoses, etc.) for use in high-pressure gaseous oxygen. A typical test system used for this testing is illustrated in Fig. 1. Although the method is commonly referred to as an "adiabatic compression" test, this paper shows that it is non-adiabatic and that substantial heat transfer occurs even for rapid pressurization.

These systems are designed to subject a material test sample or an assembled component (valve or regulator, etc.) to rapid pressure surges of gaseous oxygen from ambient pressure to a preselected test pressure through a connecting tube or tubing conduit. Tubing conduits are chosen to be consistent with the system sizes common to the material or component under test. Typical tubing conduits are either 1,000-mm-long by 5-mm internal diameter (ID) or 750-mm-long by 14-mm ID.

The pressure surge is usually required by international test standards to occur within 15–20 ms, which is achieved by rapidly opening a high-speed, high-flow, impact valve. The rapid pressurization causes an associated temperature rise in the

FIG. 1 Illustration of typical ASTM G74 test system.



gas being compressed and, if sufficiently elevated, ignition may occur on non-metallic materials (seats, seals, lubricants, contaminants, etc.). The ignition thresholds provide a way of ranking non-metallic materials and qualifying assembled components for use in oxygen at certain service pressures (including appropriate factors of safety). Compression heating of oxygen under field service conditions is widely considered an efficient ignition mechanism. Therefore, the test described earlier has gained a large subscription worldwide.

As with many ignition tests, gaseous fluid impact testing is inherently probabilistic [2] and subject to variations that are best analyzed through suitable statistical approaches. This fact has been widely discussed in the literature related to gaseous fluid impact testing [2–10]. In 1982, ASTM G74 had originally considered a "*passing*" result to be zero ignitions in 20 samples tested. Hirsch summarized the statistical considerations of this approach, "An analysis of the cumulative binomial probabilities for the ASTM G74 procedure indicated that for a probability of reaction of 0.05, assumed for a single trial, the probability of obtaining zero reactions in the 20 trials prescribed by the standard logic is about 36 %. As a result, the lack of precision with the ASTM G74 test logic could be potentially misleading when results were used to qualify materials for oxygen service" [10].

Probabilistic Nature of Ignition Testing

To illustrate the statistical variability associated with gaseous fluid impact testing, **Table 1** was developed after subjecting 20 disc samples of a polytetrafluoroethylene (PTFE) material to ignition testing by ASTM G74 using a Bruceton up/down procedure. All tests were conducted according to the precision and accuracy (Section 12) provisions of ASTM G74. This procedure requires that the test pressure be increased upon achieving a passing (no-ignition) result on a test sample and decreased upon achieving a failing (ignition) result on a test sample. These results are typical for ignition tests, and a similar pattern of ignition/no-ignition results occurs for every material tested by this method. Indeed, the behavior shown in **Table 1** has been observed at every test laboratory that has participated in the round-robin testing required by the ASTM standard. The results shown in **Table 1** seem randomly distributed and show that passing (no-ignition) and failing (ignition) results occur at many of the same pressure levels. The scatter in the results is difficult to interpret without a formal statistical analysis of the data.

The data in **Table 1** only seem randomly distributed and, with suitable statistical analysis, the ignition trends in the data can be discerned and a confidence interval established. The current version of ASTM G74 [1] provides guidance on how the data can be statistically analyzed. To illustrate the approach, disc samples of the same PTFE material were subjected to similar ignition testing; the results shown in **Fig. 2** and **Fig. 3** were obtained on the same PTFE material using the ASTM G74 test logic. The material used for this testing was a DuPont G400-7A PTFE conforming to ASTM D4894 [11]. All samples were tested in the disc configuration as specified

TABLE 1 Typical results of gaseous fluid impact testing on a PTFE material.

Pressure (bar-g)[a]	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
73																				
83		P																		
93	F		P																	
103				P																
114					P		P		P								P		P	
124						F		F		P						F		F		F
134											P		P		F					
145												F		F						
155																				
165																				

[a]The pressure values represent conversion (with rounding) to bar-g from the test pressure conducted in psi-g; P = passing result (no-ignition); F = failing result (ignition). The PTFE material tested in this series exhibited an autogenous ignition temperature, by ASTM G72, of 471°C.

FIG. 2 PTFE material testing results by ASTM G74 (logistic regression analysis). Testing by the 5-mm test system.

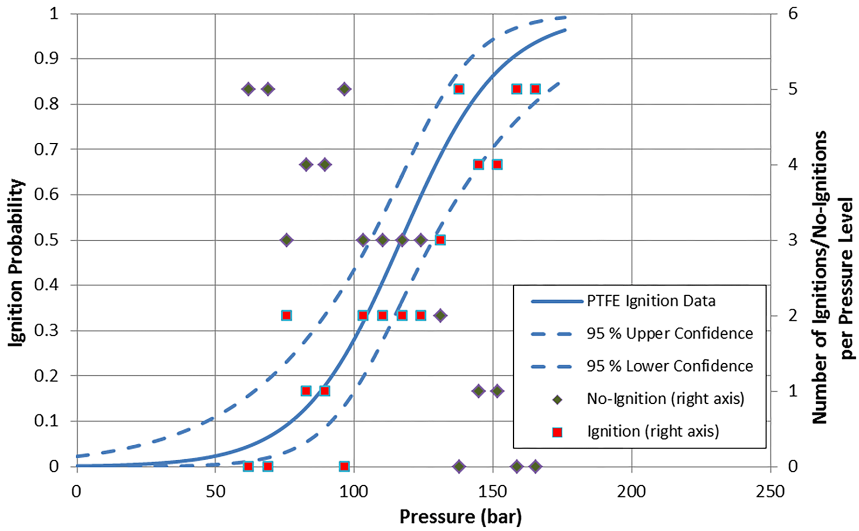
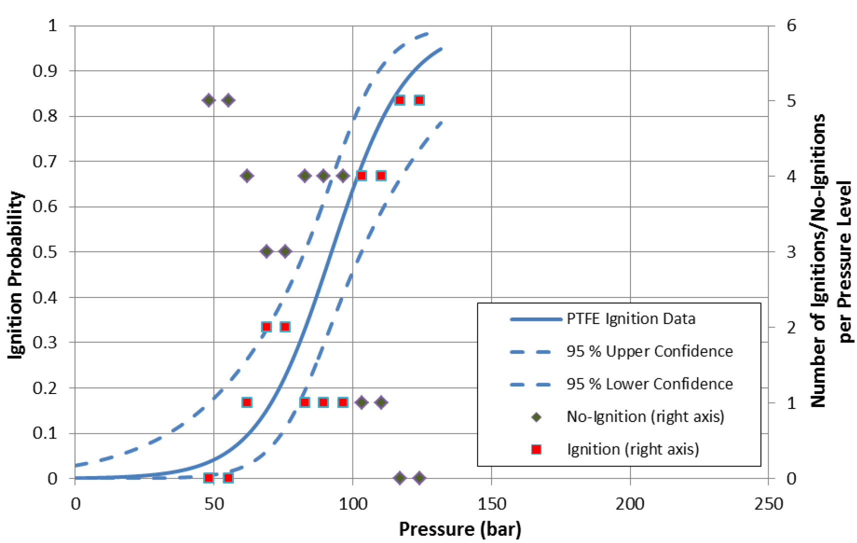


FIG. 3 PTFE material testing results by ASTM G74 (logistic regression analysis). Testing by the 14-mm test system.



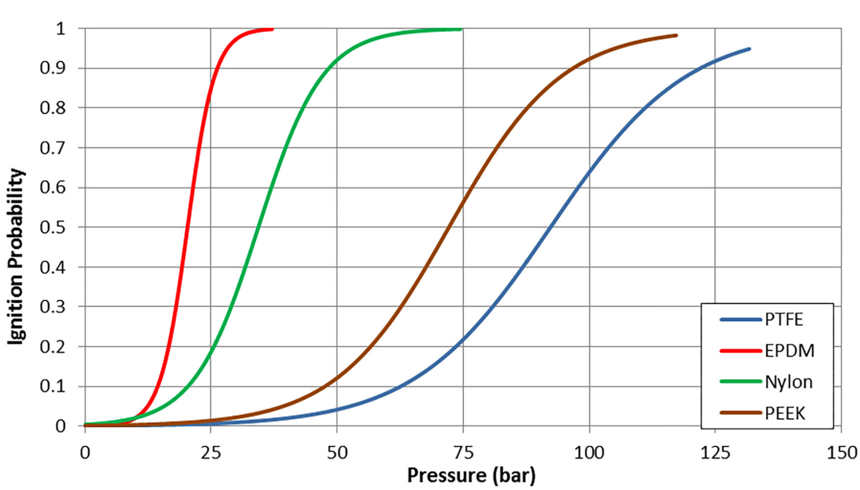
in ASTM G74. These figures show the number of ignitions (red points) and the number of no-ignitions (green points) obtained at various pressure levels. These data are well treated using an established approach for logistic regression, as has been observed and applied by several authors [2,7].

The logistic regression approach is one of the most commonly used binomial models and converts yes/no (ignition/no-ignition) data into a probability. For this testing, the probability is based on the number of ignitions and no-ignitions at each pressure level and is fitted to a standard logistic sigmoidal curve having the functionality shown in Eq 1. The beta values (β_0 and β_1) are the intercept and slope values for the function and produce the shape parameters for the sigmoidal curve and can be obtained from any number of available statistical packages. The sigmoidal curves shown in Fig. 2 and Fig. 3 are an output of a logistic regression analysis based on the specific test criteria of the different tube configurations and illustrate a way of comparing one material to another, as shown in Fig. 4. A material having higher ignition likelihood would shift the curve to the left; conversely, the curve will shift to the right for a lower ignition likelihood.

$$P(x) = \frac{e^{\beta_0+\beta_1\cdot x}}{1+e^{\beta_0+\beta_1\cdot x}} \tag{1}$$

Comparing Fig. 2 to Fig. 3 illustrates that the 14-mm system clearly produces a higher severity pressure surge than does the 5-mm system, even though each system achieves full pressurization in 15–20 ms. This is easily explained by a heat transfer

FIG. 4 ASTM G74 (14-mm system) comparison of four different disc-shaped test materials. (EPDM = Ethylene propylene diene monomer; Nylon = Polyamide Nylon 6/6; PEEK = Polyetheretherkeytone).



analysis of the impact tube. The 14-mm impact tube exhibits a lower surface area-to-volume ratio for the oxygen undergoing compression. Due to the increased diameter of the 14-mm impact tube, and the associated larger mass of oxygen undergoing compression heating while exposing a smaller perimeter heat transfer area, less heat is lost from the oxygen being compressed. Further, the larger mass achieves a higher temperature due to the lower heat rejection, and it also achieves higher internal energy (Joules) for transfer to the material test sample. Therefore, ignition testing with the 14-mm system shifts the sigmoidal curve to the left, indicating a lower pressure is required to achieve the same probability of ignition.

The value of the logistic regression approach can also be observed from Fig. 4, which allows a ranking of different materials (disc-shaped test samples) from the shape and location of the logistic sigmoidal curve and indicating the relative sensitivity of each material to this ignition method. Materials exhibiting better oxygen compatibility show a shifting of the ignition probability to the right. The least compatible material, ethylene propylene diene monomer (EPDM) in this case, exhibits the lowest pressure required before the ignition propensity starts to rise.

Another common approach is to compare materials at a specific ignition probability, such as the pressure at which 50 % of the samples ignite. ASTM G74 also describes how the 50 % reaction pressure can be developed and calculated with 20 samples (or sometimes less) if a rigorous approach, such as the Bruceton up/down procedure, is followed. As can be seen from Fig. 4, simple comparison of the 50 % reaction pressure would clearly discriminate these materials. This approach does not provide as much useful information as the logistic regression approach that was used to develop Fig. 4, but it has the advantage of requiring fewer samples to be tested for material comparisons.

Fig. 5 and Fig. 6 further demonstrate that expressing the results from ASTM G74 testing by an ignition probability based on a logistic regression is a powerful way to manage the statistical variability.

These figures demonstrate other meaningful ways to analyze the results from this type of ignition testing. Fig. 5 shows the results of conducting ASTM G74 testing on PTFE disc-shaped samples at 155 bar but varying the pressurization time rather than the pressure. In this case, the sigmoidal shape shows that the highest ignition probability occurs for the lower pressurization times, as would be expected because the compressed gas would lose less energy during the more rapid pressurization times. This figure also shows that as the pressurization time increases, the ignition probability decreases—for a given pressure, so that by 340 ms, the ignition probability is less than 10 %. This provides a way to evaluate the behavior of a material against actual service parameters for systems where pressurization rate can be controlled.

Fig. 6[4] demonstrates that the configuration of the test sample influences the results of the ignition testing such that more finely divided samples will tend to shift

[4]The data for Fig. 6 on the divided samples was provided by the German Federal Institute for Materials Research and Testing (Bundesanstalt für Materialforschung [BAM]) as part of their research during the ASTM round-robin testing on PTFE.

FIG. 5 Ignition probability of PTFE disc samples as a function of pressure rise time at 155 bar-g.

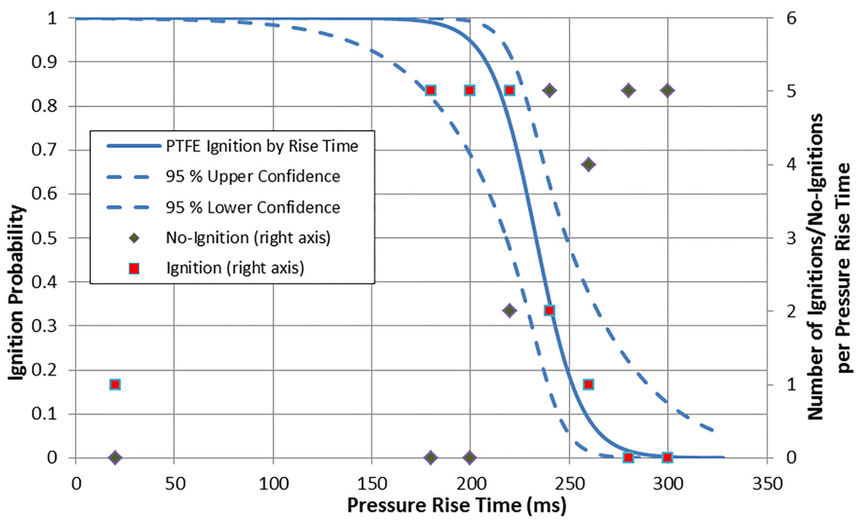
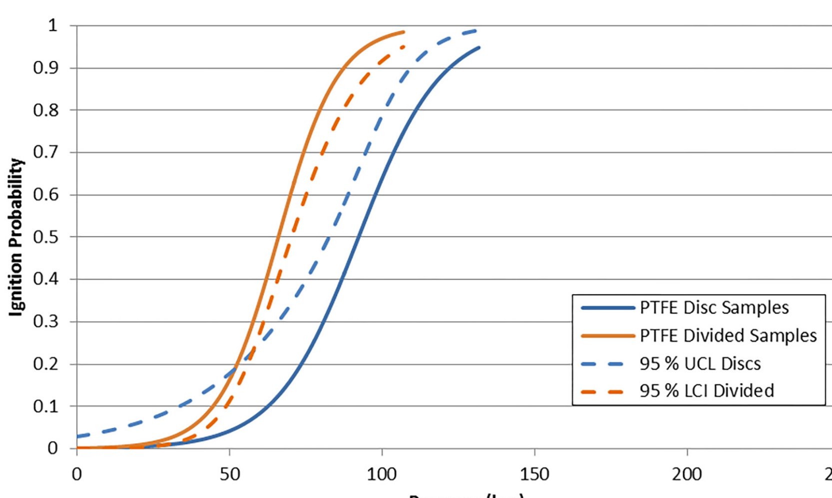


FIG. 6 Ignition probability of PTFE disc samples compared to PTFE divided samples. (NOTE: For readability, only the UCL was plotted for disc samples and the LCL for divided samples.).



the sigmoidal curve to the left, indicating a higher ignition propensity for a given pressure. Thus, divided samples that expose more surface area to the compressed gas tend to ignite more easily. This result is consistent with many of our fire investigations where samples that have a very rough surface morphology and exhibit finely divided material tend to ignite more easily than materials that maintain their initial molded surface (skin) or shape.

The figures here demonstrate that ignition testing is probabilistic and that a rigorous statistical approach such as that described herein, or equivalent, be utilized to describe and compare the ignition behavior of a material or a component for this type of testing. The authors have previously published on the background of this test method [3,8] and on a methodology for specifying the severity of the test [7,9], which was considered in the calculations discussed herein.

Despite extensive discussion of the probabilistic nature of gaseous fluid impact ignition tests in the literature, the predominant international test standards have not, to date, incorporated rigorous statistical approaches into the test requirements and still only require a low number—typically less than 10 to 20 successive pressure surges—to qualify materials and components for oxygen service. One such heavily relied upon standard, ISO Standard 21010 [12], only requires that the test pressure be reduced for each sample ignition until one sample exhibits a no-ignition result after five successive pressure surges. Once a single sample successfully passes (i.e., does not ignite) after exposure to five successive pressure surges,[5] that pressure is considered a maximum working pressure threshold. Experience indicates that, in many cases, only a few samples (<10) are tested before the no-ignition result is obtained; at which time, the testing is stopped and a pressure threshold is declared. Some laboratories continue to repeat testing until two successive samples exhibit the no-ignition result. Clearly, as indicated by **Table 1**, **Fig. 2**, and **Fig. 3**, the probabilistic nature of the ignition testing should be considered by any test logic.

The lack of attention to the statistical implications of the methodology is due in part to a predominant belief that the test is far more severe than actual service conditions. It has long been assumed that because the pressure surge occurs so rapidly (15–20 ms), the opportunity for heat rejection from the system is minimized and the test is close to adiabatic. The industry has widely described this test as the *adiabatic compression test* indicating that it was considered adiabatic. Further, the predominantly utilized temperature calculation for the final gas temperature in this test is that used to describe "isentropic compression" of an ideal gas. However, the research described herein demonstrates that the test currently being performed, at all laboratories that have participated in the ASTM round-robin testing conducted by the authors, is far from isentropic (i.e., reversible and adiabatic). However, the

[5]Every sample tested for the previous figures was also subjected to five successive pressure surges before the sample was removed to determine whether ignition had occurred. Thus, the requirement for five successive no-ignition results on a single test sample does not change the probabilistic scatter exhibited by the test.

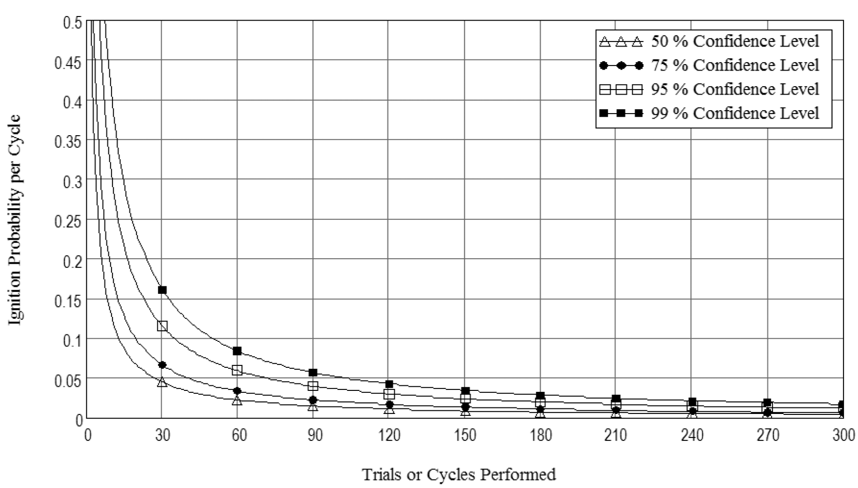
actual severity of the test has been difficult to establish due to the great difficulty in actually measuring the temperatures achieved during such a rapid pressurization event. The research described previously by the authors [7,9] to establish an approach to specifying the real severity is summarized here in order to allow a comparison of the test to service conditions.

Conclusions Regarding the Statistical Nature of ASTM G74 Testing

The previous discussion demonstrates that because the ASTM G74 test and all international tests of a similar nature use the same test embodiment, they are inherently probabilistic and subject to variability that seems random without application of appropriate statistical analysis. However, meaningful results can be developed when the appropriate statistical tools are utilized, as shown in **Fig. 2** through **Fig. 5**. Logistic regression analysis is only one available method for analyzing binomial data (ignition/no-ignition); but, it is a powerful tool that can help bring clarity to the trends in data that are obscured by seemingly random behavior, such as in **Table 1**.

Experience indicates that ignition testing where the stimulus energy is near that required for ignition (i.e., near the AIT or near the minimum ignition energy [MIE]) of a material, then the probabilistic effects are greater. When the stimulus energy is well above the AIT or the MIE, then ignition becomes more frequent for each test trial. Logistic regression provides a method of evaluating many ignition parameters while rigorously treating the statistical variations. As shown herein,

FIG. 7 Ignition probability based on binomial distribution.



many variables could be studied as was demonstrated by comparisons between disc and divided samples or ignition probability versus pressure compared to ignition probability versus pressure rise time, at a given pressure.

Ignition testing by gaseous fluid impact results in only two possible outcomes (ignition or no-ignition), and the binomial distribution, as was shown by Suvorovs, Ward, Steinberg, and Wilson [13] (**Fig. 7**), produces an undeniable confidence interval that is heavily dependent on the number of samples. This figure shows that, unless the test is very severe, which is addressed in other papers by the authors [7,14], a high number of cycles must be conducted to reach a 95 % or 99 % confidence interval. Much of the literature suggests 0.05 ignition likelihood for this testing, which would suggest then approximately 70 cycles for a 95 % confidence and 105 cycles for a 99 % confidence. Test standards and criteria that attempt to establish a threshold behavior of either a material or a component, with just a few samples or cycles, must recognize that their data may be subject to low confidence, as has been observed herein and by many previous researchers.

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